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Patent
Case No.: 55282US010

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

First Named Inventor:	MCGURRAN, DANIEL J.		
Application No.:	09/633911	Group Art Unit:	1773
Filed:	August 8, 2000	Examiner:	Paulraj, Christopher
Title:	COLOR STABLE PIGMENTED POLYMERIC FILMS		

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

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27 Aug. 2003 — Date
Signed by:

AFFIDAVIT OF DANIEL J. MCGURRAN UNDER 37 C.F.R. § 1.132

STATE OF ALABAMA)
) ss.
COUNTY OF MORGAN)

I, Daniel J. McGurran, being duly sworn, depose and say:

I.

1. That I received a Bachelor of Science degree in Chemical Engineering from the University of Minnesota in June of 1998.

2. That from 1994 to the present, I have been employed by 3M Company (hereinafter "3M", and formerly known as Minnesota Mining and Manufacturing Company), St. Paul, Minnesota in the following capacities:

(1994-1998) Technical Aide (part time);

(1998-2000) Product Development Engineer;

(2000-2002) Advanced Product Development Engineer;

(2002-2003) Senior Product Development Engineer;

(2003- present) Six Sigma Black Belt.

3. That my current position is Six Sigma Black Belt for 3M's Specialty Film and Media Products Division.

4. That I am a named inventor in the above-cited patent application.

II.

5. That I am familiar with the contents of the above-cited patent application. The pigmented films exemplified in that application are made by a process in which particulate pigment is added to a reaction mass before polymerization.

6. That I have read U.S. Patent No. 4,865,898 (Fukuda et al.). The pigmented films exemplified by Fukuda et al. are made by a process in which particulate pigment is added to an already polymerized polyester.

7. That I have recently had two pigmented films produced under my direction for the purpose of demonstrating the effect of adding the particulate pigment to an already polymerized polyester, compared to adding the particulate pigment to the reaction mass before polymerization, on the haze of the final film.

III.

8. That a first pigmented film (the "First Film") was produced under my direction by a process in which carbon black particles were kneaded into an already polymerized polyester.

9. That the First Film was made according to the following procedure. First, a quantity of pellets of an already polymerized standard commercial film-grade

polyethylene terephthalate (PET) resin made by 3M, substantially free of any particulate matter such as carbon black or slip agents, was obtained. A carbon black pigment (Cabot Black Pearls 1300, with a stated 13 nm average particle size) was also obtained. This carbon black material is the same type used to make the low haze films of Examples 7-12 in the above-cited patent application. The pellets were metered at 39.1 kg/hr (86.3 lbs/hr) into the feed throat of a 70 mm Werner-Pfeiderer twin screw extruder that was equipped with three kneading blocks and set up in a configuration known to be effective for mixing. The carbon black was metered at 0.318 kg/hr (0.7 lbs/hr) (so as to make a final film having a carbon black loading of 0.8 wt%) into the atmospheric port of this extruder in the third of eight extruder barrel zones. This feed location is believed to be the optimal feed location for achieving good mixing of a powder into the polymer on this equipment. The extruder was run at 47 rpm. Zone temperature set-points were 38 °C (100 °F), 116 °C (240 °F), 154 °C (310 °F), 232 °C (450 °F), 263 °C (505 °F), 271 °C (520 °F), 274 °C (525 °F), and 274 °C (525 °F), working from the feed end of the kneader-extruder to the die end. The melt temperature near the die end was measured to be about 283 °C (542 °F). The melt was metered through a 20 micron metal filter. A standard film "drop" die was used, and the web was cast at about 539 cm/min (17.7 feet/min) onto a water-cooled casting roll. The cast web thickness was about 0.22 mm (0.0085 inches), and the width was about 42 cm (16.5 inches). Squares 11.5 cm (4.5 inches) on a side were cut from the cast web and biaxially oriented using a laboratory stretching apparatus, model Karo IV manufactured by Bruckner. Multiple samples of the First Film were obtained by stretching each square of cast web to a draw ratio of 3.5 in the machine direction by 3.5 in the transverse direction, using conditions generally found to be optimal for PET films on this stretching apparatus.

10. That the First Film was measured to have the following properties:

Thickness: 19.1 μm (0.00075 inches) (average of 6 samples, measured with a Mitutoyo film thickness gauge)

Transmission: 33.4%	(average of 6 samples, measured with a Gardner haze-gard plus system)
Total Haze: 42.5%	(average of 6 samples, measured with the Gardner haze-gard plus system)
Internal Haze: 14.7%	(measured in the same way as Total Haze except with an index matching fluid to eliminate surface haze)

11. A sample of the First Film is attached hereto as Exhibit A.

IV.

12. That a second pigmented film (the "Second Film") was produced under my direction by a process in which carbon black particles were kneaded into a reaction mass before polymerization. The composition and thickness of this film were substantially the same as those of the First Film.

13. That the Second Film was made according to the following procedure. First, a quantity of pellets of a pigmented masterbatch was produced on an industrial production scale by 3M using a procedure similar (but not identical) to the laboratory- or pilot-scale procedures outlined in the Examples section of the above-cited patent application. Specifically, PVP (as a dispersing agent) was blended into ethylene glycol, and Cabot Black Pearls 1300 carbon black powder was added. The mixture was processed in a Cowles dissolver. The mixture was then passed multiple times through a vertically-oriented Netzsch sand mill, having a 50% by volume loading of 1.0-1.25 mm ceramic media, in order to achieve an adequate degree of dispersion. No subsequent filtration was employed. This dispersion and additional ethylene glycol was charged to a production-scale batch reaction kettle. Di-methyl terephthalate and catalyst was added and the reaction mass was polymerized to a carbon-black loaded PET masterbatch under typical PET reaction conditions.. Note that this masterbatch incorporated the very same carbon black particles that were used in the manufacture of the First Film. The masterbatch contained 1.5 wt% carbon

black. Next, a quantity of pellets of an already polymerized standard commercial film-grade polyethylene terephthalate (PET) resin made by 3M, substantially free of any particulate matter such as carbon black or slip agents, was obtained. These non-pigmented pellets were the same as those used in the manufacture of the First Film. The masterbatch pellets were metered at 21.8 kg/hr (48 lbs/hr), and the polyester pellets were metered at 19.1 kg/hr (42 lbs/hr), both types of pellets going into the feed throat of the same 70 mm Werner-Pfeiderer twin screw extruder referred to above. This resulted again in a final film having a carbon black loading of 0.8 wt%. The extruder was again run at 47 rpm, and the zone temperatures were the same as for the production of the First Film. The melt temperature near the die end was measured to be about 281 °C (538 °F). The melt was metered through a 20 micron metal filter. The same standard film "drop" die was used, and the web was cast at about 549 cm/min (18.0 feet/min) to a water-cooled casting roll. The cast web thickness was about 0.22 mm (0.0085 inches), and the width was about 42 cm (16.5 inches). Squares 11.5 cm (4.5 inches) on a side were cut from the cast web and biaxially oriented using the Bruckner model Karo IV laboratory stretching apparatus. Multiple samples of the Second Film were obtained by stretching each square of cast web to a draw ratio of 3.5 in the machine direction by 3.5 in the transverse direction, using same conditions referred to above in connection with the First Film.

14. That the Second Film was measured to have the following properties:

Thickness: 18.5 μm (0.00073 inches) (average of 6 samples, measured with a Mitutoyo film thickness gauge)

Transmission: 19.7% (average of 6 samples, measured with the Gardner haze-gard plus system referred to above)

Total Haze: 15.2% (average of 6 samples, measured with the Gardner haze-gard plus system)

Internal Haze: 6.2% (measured in the same way as Total Haze except with an index matching fluid to eliminate surface haze)

15. A sample of the Second Film is attached hereto as Exhibit B.

16. The undersigned declares that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Further Affiant Saith Not.

Daniel J. McGuiran

Subscribed and sworn to before me
this 21th day of August, 2003.

Notary Public

(Seal)

Exhibit A: "First Film"

Exhibit B: "Second Film"